



October 01, 2015

Tom Moe USS Corporation P.O. Box 417 Mountain Iron, MN 55768

RE: Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on September 23, 2015. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Heather R Zika

Haller Zto

heather.zika@pacelabs.com

Project Manager

Enclosures

cc: Terri Sabetti, Northeast Technical





Pace Analytical www.pacelabs.com

315 Chestnut Street Virginia, MN 55792 (218) 742-1042

CERTIFICATIONS

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792 Alaska Certification #MN01084 Arizona Department of Health Certification #AZ0785 Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification #: 998027470 WA Department of Ecology Lab ID# C1007 Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

315 Chestnut Street Virginia, MN 55792 (218) 742-1042



SAMPLE SUMMARY

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1254030001	WS-002 Scrubber Make-Up	Water	09/23/15 10:15	09/23/15 17:00
1254030002	WS-003 Thickener Overflow	Water	09/23/15 10:15	09/23/15 17:00



SAMPLE ANALYTE COUNT

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1254030001	WS-002 Scrubber Make-Up	EPA 200.7	MAR	3	PASI-V
		EPA 300.0	CSD	1	PASI-V
1254030002	WS-003 Thickener Overflow	EPA 200.7	MAR	3	PASI-V
		EPA 300.0	CSD	1	PASI-V



ANALYTICAL RESULTS

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Date: 10/01/2015 04:43 PM

Sample: WS-002 Scrubber Make	-Up Lab ID:	1254030001	Collecte	d: 09/23/1	5 10:15	Received: 09/	23/15 17:00 Ma	atrix: Water	
			Report						
Parameters	Results	Units	Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
200.7 MET ICP, Lab Filtered	Analytical	Method: EPA	200.7 Prepa	aration Meth	od: EP	A 200.7			
Calcium, Dissolved	88.6	mg/L	5.0	0.29	10	09/28/15 13:50	09/30/15 09:48	7440-70-2	
Magnesium, Dissolved	200	mg/L	5.0	0.67	10	09/28/15 13:50	09/30/15 09:48	7439-95-4	
Total Hardness, Dissolved	1050	mg/L	100	50.0	10	09/28/15 13:50	09/30/15 09:48		
300.0 IC Anions 28 Days	Analytical	Method: EPA	300.0						
Sulfate	736	mg/L	20.0	0.89	10		09/26/15 03:05	14808-79-8	
Sample: WS-003 Thickener Overflow	Lab ID:	1254030002	Collected	d: 09/23/1	5 10:15	Received: 09/	23/15 17:00 Ma	atrix: Water	
	Lab ID:	1254030002 Units	Collected Report Limit	d: 09/23/15 MDL	5 10:15 DF	Received: 09/	23/15 17:00 Ma	atrix: Water CAS No.	Qual
Overflow	Results		Report Limit	MDL	DF	Prepared			Qual
Overflow Parameters	Results	Units	Report Limit	MDL	DF	Prepared		CAS No.	Qual
Overflow Parameters 200.7 MET ICP, Lab Filtered	Results Analytical	Units Method: EPA	Report Limit 200.7 Prepa	MDL aration Meth	DF nod: EP/	Prepared A 200.7	Analyzed	CAS No.	Qual
Parameters 200.7 MET ICP, Lab Filtered Calcium, Dissolved	Results Analytical	Units Method: EPA 2 mg/L	Report Limit	MDL aration Meth 0.29	DF nod: EP/	Prepared A 200.7 09/28/15 13:50	Analyzed 09/30/15 09:52	CAS No.	Qual
Parameters 200.7 MET ICP, Lab Filtered Calcium, Dissolved Magnesium, Dissolved	Results Analytical 910 ND 2280	Units Method: EPA : mg/L mg/L	Report Limit 200.7 Prepa 5.0 5.0 100	MDL aration Meth 0.29 0.67	DF nod: EP/ 10 10	Prepared A 200.7 09/28/15 13:50 09/28/15 13:50	Analyzed 09/30/15 09:52 09/30/15 09:52	CAS No.	Qual



QUALITY CONTROL DATA

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Date: 10/01/2015 04:43 PM

QC Batch: MPRP/5901 Analysis Method: EPA 200.7

QC Batch Method: EPA 200.7 Analysis Description: 200.7 MET Dissolved

Associated Lab Samples: 1254030001, 1254030002

METHOD BLANK: 251582 Matrix: Water

Associated Lab Samples: 1254030001, 1254030002

ParameterUnitsBlank ResultReporting LimitAnalyzedQualifiersCalcium, Dissolvedmg/LND0.5009/30/15 08:52

Magnesium, Dissolved mg/L ND 0.50 09/30/15 08:52

LABORATORY CONTROL SAMPLE: 251583

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Calcium, Dissolved 50 51.2 102 85-115 mg/L Magnesium, Dissolved 50 51.1 102 85-115 mg/L

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 251584 251585 MSD MS 1254191001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits **RPD** RPD Qual Calcium, Dissolved mg/L 227 50 50 284 281 114 108 70-130 20 Magnesium, Dissolved mg/L 129 50 50 182 182 106 107 70-130 0 20

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 251586 251587 MS MSD 1254191008 MS MSD MS Spike Spike MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Calcium, Dissolved 50 227 50 284 280 114 70-130 20 mg/L 106 50 Magnesium, Dissolved 130 50 182 179 105 99 70-130 2 20 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

NPDES - Line 3 Weekly Project:

Pace Project No.: 1254030

QC Batch: WETA/13878 QC Batch Method: EPA 300.0

Analysis Method:

EPA 300.0

Analysis Description:

300.0 IC Anions

Associated Lab Samples: 1254030001, 1254030002

METHOD BLANK: 251256

Matrix: Water

Associated Lab Samples:

1254030001, 1254030002

Blank Result Reporting

Parameter

Units

Limit Analyzed

Qualifiers

Sulfate

mg/L

ND

2.0 09/26/15 02:43

LABORATORY CONTROL SAMPLE:

Parameter

Units

mg/L

Spike Conc.

LCS Result

LCS % Rec % Rec Limits

90-110

Qualifiers

Sulfate

Sulfate

Units mg/L

50

49.7

251259

MATRIX SPIKE & MATRIX SPIKE DUPLICATE:

251258

MSD

MS 1253877001 Spike Result Conc.

MSD Result

MS % Rec

MSD % Rec % Rec Limits RPD

Max RPD

Parameter

Spike Conc. 2.4 50 50

MS Result 52.2

52.2 100 100

90-110

0 20

Qual

Date: 10/01/2015 04:43 PM

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALIFIERS

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

Date: 10/01/2015 04:43 PM

PASI-V Pace Analytical Services - Virginia



QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES - Line 3 Weekly

Pace Project No.: 1254030

Date: 10/01/2015 04:43 PM

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1254030001 1254030002	WS-002 Scrubber Make-Up WS-003 Thickener Overflow	EPA 200.7 EPA 200.7	MPRP/5901 MPRP/5901	EPA 200.7 EPA 200.7	ICP/4604 ICP/4604
1254030001 1254030002	WS-002 Scrubber Make-Up WS-003 Thickener Overflow	EPA 300.0 EPA 300.0	WETA/13878 WETA/13878		

Required Client Information: # company: ft. Iron, MN 55768 N lequested Due Date: 9 ITEM# 8 WS-002 Scrubber Make-Up WS-003 Thickner Overflow P.O. Box 417 USS Corporation One Character per box.
(A-Z, 0-9 / , -)
Sample Ids must be unique SAMPLE ID ADDITIONAL COMMENTS MATRIX
Drinking Water
Water
Waste Water
Product
Soll/Solid
Oil
Wipe
Air
Other Purchase Order #:
Project Name: N Report To: Tom Moe Required Project Information Copy To: roject #. \$9\$\$P\$*\$\$\$ OM Moclies RELINGUISHED BY / AFFILIATION MATRIX CODE (see valid codes to left) ≤ ₹ SAMPLE TYPE (G=GRAB C=COMP) NPDES-LINE 3 Wkly DATE START AMPLER NAME AND SIGNATURE H H H **5**/ COLLECTED PRINT Name of SAMPLER The Chain-of-Custody is a LEGAL DOCUMENT. All re-DATE CHAIN-OF-CUSTODY / Analytical P-10 <u>Q</u> 8 14/16 15 TIME 15 DATE SAMPLE TEMP AT COLLECTION Invoice Information: Attention: Pace Quote: Company Name: # OF CONTAINERS Pace Project Manager: Pace Profile #: Address: 100 Unpreserved H25O4 HNO3 HCI NaOH Na2S2O3 heather.zika@pacelabs.com ACCEPTED BY JAFFILLATION Methanol Y/N Analyses Test LAB FILTERED: SO4 WO#: 1254030 CLIENT: USS CORP PM: HRZ DATE Signed: × × Lab FILTERED: Ca,Mg,Har 12/2 DATE Due Date: 10/07/15 3 THE ر الم TEMP in C Residual Chlorine (Y/N) Received on LF,LF ᄕ,ᄕ SAMPLE CONDITIONS. (Y/N) Custody Sealed Cooler (Y/N) Samples intact (Y/N)

Pace Analytical*

Project Manager Review:

hold, incorrect preservative, out of temp, incorrect containers)

Document Name:

Sample Condition Upon Receipt Form

Document No.: F-VM-C-001-Rev.09 Document Revised: 23Feb2015 Page 1 of 1

Issuing Authority:

Issuing Authority:
Pace Virginia, Minnesota Quality Office

Sample Condition **Client Name:** Project #: WO#: 1254030 **Upon Receipt** Courier: Fed Ex UPS USPS Client Pace Other:_ ■Commercial Tracking Number: Optional: Proj. Due Date. Custody Seal on Cooler/Box Present? □No ΠNo Packing Material: Bubble Wrap Bubble Bags None Other: Temp Blank? □No Thermometer Used: 140792808 Type of Ice: Wet Blue None Samples on ice, cooling process has begun Cooler Temp Read °C: Cooler Temp Corrected °C: Biological Tissue Frozen? Temp should be above freezing to 6°C Correction Factor: 0.3 Date and Initials of Person Examining Contents: Comments: Chain of Custody Present? □No □N/A Chain of Custody Filled Out? □No 2. □N/A Chain of Custody Relinquished? □No □N/A □No Sampler Name and Signature on COC? □N/A □No Samples Arrived within Hold Time? □N/A Yes ZNo □N/A Short Hold Time Analysis (<72 hr)? Rush Turn Around Time Requested? Yes □N/A Yes □No Sufficient Volume? □N/A 8. Yes Correct Containers Used? □No □N/A Yes □No -Pace Containers Used? □N/A Yes Containers Intact? □ No □N/A □N/A Filtered Volume Received for Dissolved Tests? ☐Yes **₽**No 11. Note if sediment is visible in the dissolved containers. Yes Sample Labels Match COC? □No □N/A 12. -Includes Date/Time/ID/Analysis Matrix: See pH log for results and additional preservation All containers needing acid/base preservation will be Yes □No □N/A documentation checked and documented in the pH logbook. Yes □No N/A Headspace in Methyl Mercury Container 13. ☐Yes □No M/A Headspace in VOA Vials (>6mm)? 14. Trip Blank Present? Yes □No M/A 15. Trip Blank Custody Seals Present? Yes □No Pace Trip Blank Lot # (if purchased):_ CLIENT NOTIFICATION/RESOLUTION Field Data Required? Yes No Person Contacted: Date/Time: Comments/Resolution: FECAL WAIVER ON FILE **TEMPERATURE WAIVER ON FILE**

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (I.e. out of